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National Aeronautics and Space Administration
Headquarters
Washington, D. C. 20546

Attention: Mr. J. Gangler
Code RRM

Gentlemen:

Subject: Interim Technical Report No. 1
Contract NASW-2187

Forwarded herewith in accordance with the Article VIII requirements of the contract schedule are ten (10) copies and one (1) reproducible copy of the subject report.

Very truly yours,

Allen A. Navarro

Allen A. Navarro
Contract Administrator

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Enclosures (11)

DEFORMATION PROCESSES IN FORGING CERAMICS

Progress Report No. 1

8 April 1971 - 8 July 1971

Prepared for

Office of Advanced Research and Technology
National Aeronautics and Space Administration
Headquarters
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by

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W.H. Rhodes

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FOREWORD

This work is being performed under the sponsorship of the NASA Headquarters, Office of Advanced Research and Technology, Research Division with Mr. J. Gangler as Project Monitor under Contract NASW-2187.

The work is being performed at the Avco Corporation, Systems Division Lowell, Massachusetts in the Materials Sciences Department managed by Dr. T. Vasilos. Mr. R.M. Cannon is directing the work with the assistance of Dr. W.H. Rhodes. The authors wish to acknowledge the assistance of Mr. B. MacAllister in Mechanical Testing and the microscopy of Mr. C.L. Houck and R.E. Gardner.

ABSTRACT

The program objective is to investigate the deformation processes involved in the forging of refractory ceramic oxides. A combination of mechanical testing and forging is being utilized to investigate both the flow and fracture processes involved.

During this quarter a series of stress-strain rate tests on 1.2 μ Al_2O_3 + 1/4% MgO were performed in the range of 1200-1500°C. A variable strain rate sensitivity was found which suggests a possible shift in the deformation mechanisms; however, interpretation has been delayed until further verification of all the effects obtained. In addition, another deep drawn hemisphere of alumina was attempted at a lower temperature.

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I. INTRODUCTION

The objective of this program is to investigate the forgability of the refractory oxides. The approach taken includes an investigation of the necessary high temperature deformation and fracture behavior of these materials in order to provide information and understanding which can then be applied directly to forging problems. The primary emphasis has been on mechanical properties studies. A few forgings are being done to supplement the mechanical testing results.

The report of the first year of work included reviews of the work and understanding to date of the high temperature mechanical behavior of the oxides as well as hot working efforts with these materials.[1] On the basis of this study, two systems were identified for primary investigation. These were fine-grained alumina, doped to inhibit grain growth, and magnesia. For alumina, retention of a relatively fine grain size is important even at very high temperatures in order to obtain adequate ductility. For magnesia at temperatures of 2100°C or above, adequate ductility from slip processes was indicated as probable.

During the first year's effort extensive mechanical testing of fine-grained alumina was performed to clarify the contributing mechanisms of deformation at very fine grain sizes, to provide further data on flow stresses for use in forging, and to indicate the origins of cracking and cavitation at grain boundaries. In addition, a few forgings were done to correlate with the mechanical test results.

Considerable evidence was found for increasing contributions of non-Newtonian deformation processes including grain boundary sliding and dislocation motion was found for grain sizes below 5 μ. In addition, some unexplained effects of specimen purity or test atmosphere on the flow stress were indicated.

Considerable capacity for deformation was demonstrated although several causes of cracking were indicated. For most of the flexural tests and the forgings, the limiting cracks occurred at defective areas in the specimens which included coarse grained patches, pore nests and regions of impurity concentration which may also contribute to the other two problems. In addition, cavitation at grain boundaries also develops and presumably would be the limiting feature if the defective regions were eliminated in the specimens. The appearance and growth of cracks was shown to be faster at higher strain rates with the associated higher stresses.

Further investigation of some of the unresolved problems is being undertaken during the present program. Additional forgings will also be performed as indicated appropriate by the mechanical test results. The effect of specimen purity or test atmosphere on the flow stress is particularly important to the forging effort since it is desirable to achieve high strain rates at low stresses and without development of internal cracks. Investigation of the actual origin of the defect regions is beyond the scope of this program; however, another program[2] is presently investigating the origin of these flaw areas and the results will be used to obtain improved specimens for this work.

During the past year on another program[3] some very valuable comparative data were obtained on the deformation behavior of higher purity alumina. The material which had been specially made had a grain size of 1.2 μ which made it almost identical to the C126C alumina tested on last year's program[1] except that it had a somewhat lower total impurity content and was not doped with 0.25% MgO as was that tested on this program. The results were extremely interesting in that the high purity materials had nearly comparable flow stresses to the C126C data where comparable test conditions existed, but the high purity material exhibited generally higher values of rate sensitivity with m values between 0.77 and 0.93 in the range of 1228-1400°C compared to values of 0.65 to 0.71 for the C126C specimens. The second difference was that higher flow stresses could be achieved before specimen fracture became a limiting feature.

Because of the valuable insight to be gained from the comparative information, additional tests of C126C specimens were performed during this reporting period in order to provide further data under test conditions comparable to those used for the high purity specimens. Also, during this quarter, an additional hemisphere forging was attempted and microstructural evaluation of this and the previous hemispheres was initiated.

II. MECHANICAL TEST RESULTS

As indicated in the introduction, additional tests of the C126C Al_2O_3 + 1/4% MgO were performed to provide stress-strain rate relations over an extended range of temperature and strain rate. This was initiated to provide more direct comparison with data on high purity alumina tested on another program[3]; however, as the results showed some unexpected behavior, the test schedule was extended to investigate these effects.

The testing was done in four-point flexure in an argon atmosphere furnace. The load is recorded continuously versus time. Deflection is measured with a probe system monitored with an LVDT which is also continuously recorded in order to be able to determine stress, strain and strain rate continuously throughout the test. The tests were primarily run at a series of strain rates by holding the rate constant until a steady-state load was achieved and then increasing the rate and again waiting until steady-state conditions were obtained before repeating the procedure. For most specimens the maximum strain was limited to 2-3% to keep spurious effects from large curvature[1] to a minimum. The test machine is a constant cross-head machine with a variable speed drive. Considerable effort was expended to obtain data at the lowest possible speeds achievable on the unit.

The C126C specimens are from the same hot pressed billet as those previously tested; the material was made from a standard 99.9+% pure, 0.3 μ grade alumina powder with an addition of 1/4% MgO for grain growth inhibition during pressing and testing. The grain size measured on specimens after testing was 1.2-1.3μ* except for some of the higher temperature tests which are discussed below. The high purity specimens had been hot pressed from a special, high purity powder without any additives; the grain sizes averaged

* The grain sizes were all measured by the linear intercept method and reported as $G = 3/2\bar{L}$.

about 1.2 μ, although testing could not be done at 1400°C or above without significant grain growth.[3]

The test results are plotted in Figure 1 as log stress versus log strain rate. This plot includes the data previously reported[1] plus the recent tests. The stresses have been calculated using the elastic approximation:

$$\sigma = \frac{3\ Pa}{bh^2} \qquad (1)$$

Correction for plastic bending and large curvature effects will be made after the sequence has been completed and the cause of the several effects established. Also shown on the plot are the lines for the high purity tests at temperatures of 1228, 1258, 1288 and 1337°C.

These curves show a decided effect of the actual strain rate upon the strain rate sensitivity m_b given by

$$m_b = \frac{\partial \log \sigma}{\partial \log \dot{\epsilon}}$$

The curvature in the plots become more obvious by extending the range of strain rate and temperature of the tests. It can be seen that the results of the high purity specimens are very similar to the C126C specimens and that the difference in apparent values of m_b was more a function of the range of stress-strain rate over which the measurements were made.

Because of the obvious implications of the similarity of results for different purity levels and of the variation of the rate sensitivity, considerable effort was expended to ensure that the results were reproducible and not test artifacts. The break in the low temperature curves which shows an increase in slope at higher stress and strain rate is reproducible and does not seem to be due simply to a systematic test artifact. None of the previous work[1,4] on fine-grained alumina covered a sufficiently broad range of strain rate to demonstrate curvature conclusively. It can be seen, however, that some of the variation in m_b with temperature previously reported is consistent with these results.

The second break seen only in the higher temperature curves cannot be held in high confidence without further verification. The problem is that at the long times necessary for the low rates appreciable grain growth can occur at the higher temperatures. Grain sizes as high as 2 μ were found in some of the specimens after testing at 1490°C or after very long tests at 1418°C. Because of the high dependence of strain rate on grain size, it is possible that the high initial slope may be a consequence of grain growth during the early part of the test. A parabolic or cubic grain growth law would result in a greater effect during the early part of the test, i.e., on the lower strain rate points. As a result of these uncertainties,

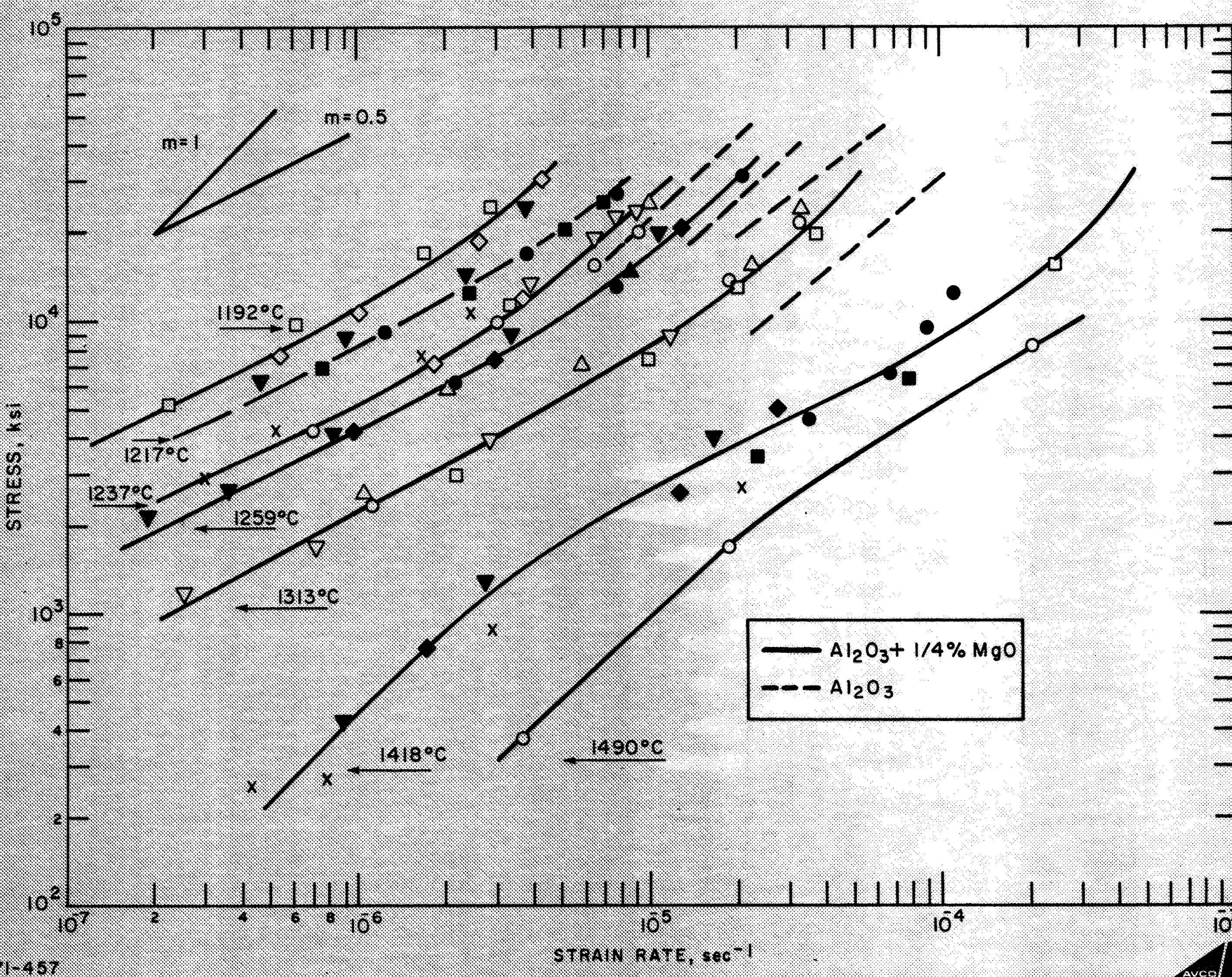

Figure 1. Stress-Strain Rate Curves for Fine Grained Al_2O_3

some further tests are being conducted to determine whether this break is real or an artifact from grain growth.

These test results have some rather interesting implications regarding the deformation mechanisms. A preliminary plot of strain rate versus temperature indicates that there is no significant difference in the high purity specimens and the C126C specimens for comparable stress and temperature ranges. This in itself is rather surprising. The C126C specimens not only have a somewhat higher impurity content, but contain 1/4% MgO. Because of presumed segregation of the MgO near grain boundaries, it seems quite surprising that there would be no effect on a mechanism involving grain boundary sliding.

In addition to the negative effect of purity, the change in rate sensitivity is also surprising. No similar behavior of increasing sensitivity with increasing strain rate for ceramic materials is known to the authors. Somewhat similar behavior has been reported for some of the superplastic metals investigated[5,6]; for these cases, however, the m values increased from about 0.2 to a maximum of about 0.5 which is a lower range of m than seen in the present work where values range from 0.5 up to nearly unity.

Further interpretation of these curves is premature at this time without better verification of the apparent high slope region at low strain rates. If this is definitely established then it would be expected at the lower temperatures at sufficiently low rates. Since interpretation will depend significantly on the existence of this second break, explanations would be premature at this time. It seems likely that understanding of this strain rate dependence should allow a better assessment of the relative contributions of non-Newtonian processes such as slip and Newtonian viscous deformation mechanisms such as diffusional creep. It should also be mentioned that transient effects could perhaps contribute to this observed behavior and will be considered in evaluation and interpretation of these data.

III. FORGING

An attempt was made to deep draw a hemisphere from a flat blank of alumina at 1575°C. The blank had an initial grain size of 2.4 μ. A new punch and die were used. The die had an entrance angle of 30° and a small entrance radius compared to the previously used set-up[1] which had a 40° entrance angle and generous radius. The forging, D1600, was done at 1575°C to reduce the amount of grain growth occurring during the deep drawing.

The blank was broken into several pieces as can be seen in Figure 2. Examination of the fracture surfaces was somewhat ambiguous. The principal radial cracks clearly occurred quite early in the test, and appeared to originate in the flat part of the crack between the two largest pieces. It was not clear exactly where in this region the crack started. It seems likely that the initial crack may have resulted from a brief increase in strain rate and load during the early part of the test. A second set of cracks also developed at the entrance region where the maximum bending occurs. This appears to be the result of drawing over too sharp a radius at too high a rate.

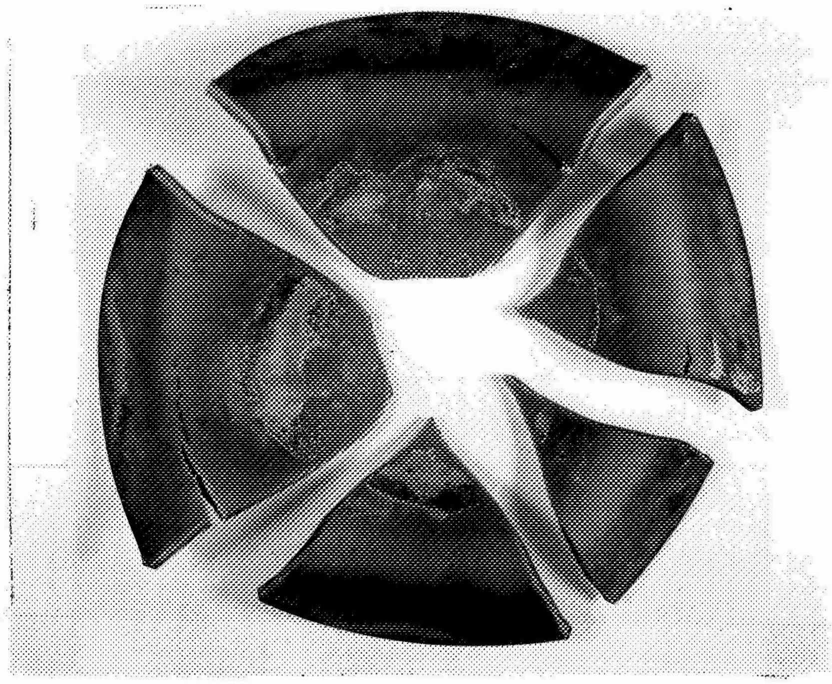

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Figure 2. Cracking of Deep Drawn Hemisphere Attempt. Forging D1600 of Al_2O_3. The upper end right pieces should be interchanged for proper order.

Examination of the surfaces of this forging indicated some chemical compatibility problems which must be avoided. Some evidence of a molten phase could be seen on the surface; this is thought to be B_2O_3 from the BN lubricant used. It is thought more likely that the oxide was initially present in the BN, although it may have formed during forging. Appropriate steps will be taken to eliminate the problem since the attack by a liquid phase could lead to cracking during forging.

IV. MICROSTRUCTURAL EVALUATION

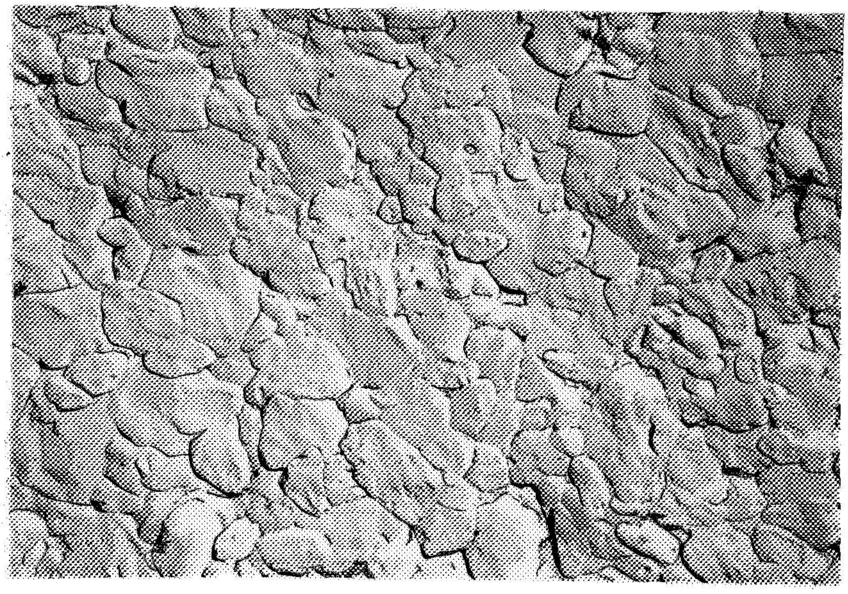
Evaluation was begun on the broken hemisphere as well as on the more successful one, done at 1625°C previously reported.[1] The previous one, D1448, was etched in phosphoric acid and the inner and outer surfaces were replicated for electron microscope examination. A typical region from the outer surface is shown in Figure 3a; it can be seen that appreciable grain growth occurred. The average was 6.8 μ compared to an initial size of 3 μ for this piece. On this piece many small spots could be visually seen; replication showed that these were small coarse-grained patches. The grains in these patches were about 30 μ as can be seen in Figure 3b. These coarse grained regions acted as hard spots and did not deform as fast as the surrounding regions. Some cracking was observed in the regions immediately surrounding the hard spots which presumably resulted from the higher local strain rates.

Examination of the pieces of D1600, done at 1575°C showed that less grain growth occurred, as anticipated. The grain size increased from 2.4 μ to aboue 3.8 μ which is desirable for the final product. This piece also had coarse grained patches in it after forging as shown in Figure 4. Because there was less thinning of this piece the coarse grained spots did not stand high on the surface as in D1448. It is presently thought that these coarse grained patches are in the original blanks although some further grain growth may occur during forging. Reduction in the occurrence of these types of defects is the object of another program[2] and the results will be used in the future to obtain improved preform blanks.

V. FUTURE WORK

During the next quarter further verification of the variable strain rate sensitivity will be sought by further testing, especially at low strain rates. Upon completion of this, final reduction and analysis of these data will be done. Additional microstructural evaluation of specimens tested at different regions of rate sensitivity will be done to aid in evaluation of these data.

Additional drawings of hemispheres are also planned. Some modifications of die design and loading procedures will be undertaken to eliminate early cracking.



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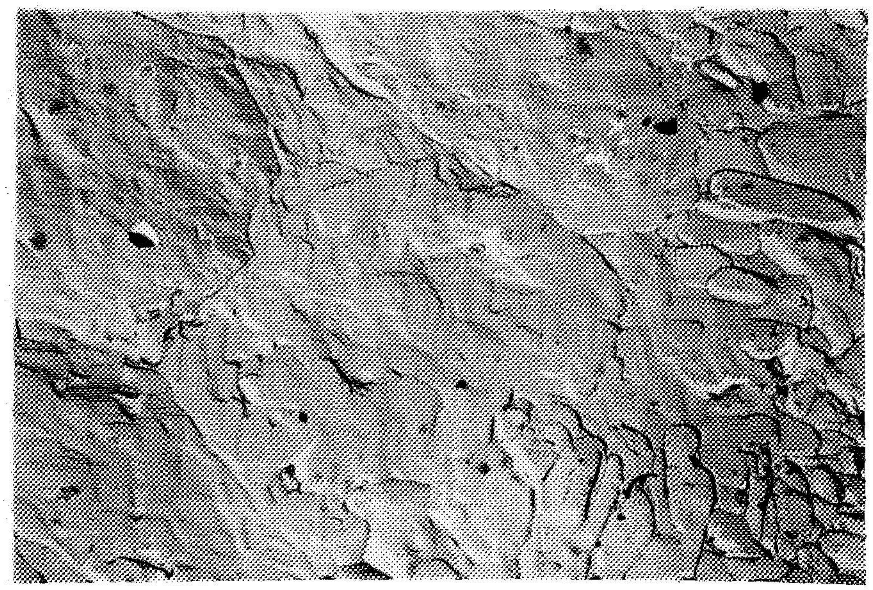

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Figure 3. Microstructure of Outside Surface of Hemisphere D1448 Showing a Typical Area (a) and an Area at the Edge of a Coarse-Grained Patch (b).

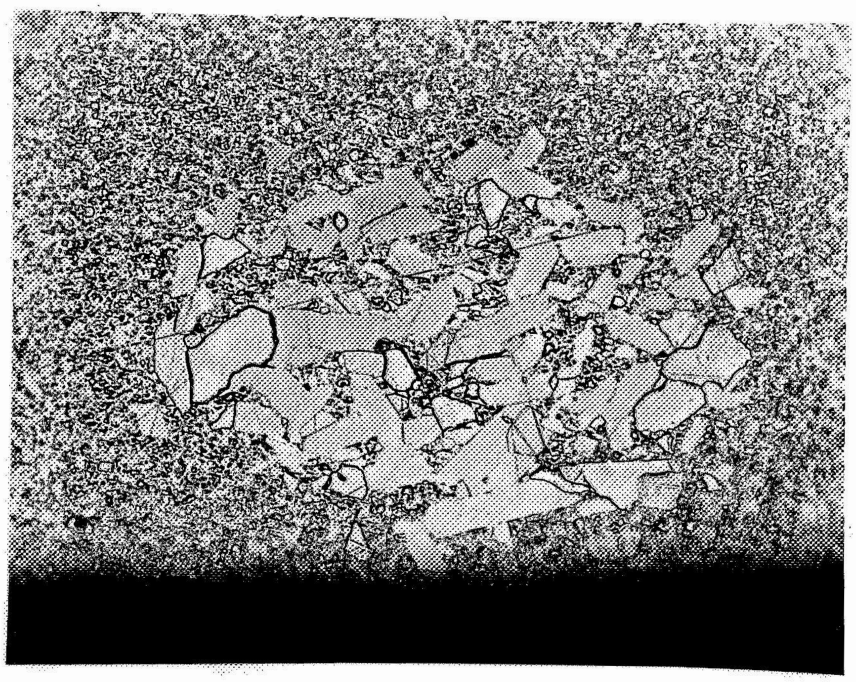

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Figure 4. Coarse-Grained Patch in Piece from Broken Hemisphere, D1600.

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